

STTR N03-T013 (Innovative Vehicle Camouflage)

Contract No. N00014-03-M-0345

Phase I Final Report

Period 2003Oct31 thru 2004 Jan30

Phase I Executive Summary:

The use of painted camouflage patterns on military hardware is a time-tested, cost-effective and practical countermeasure against human vision and aided vision target acquisition systems in many combat scenarios. However, the ability to integrate advanced patterns and optical effects is inherently limited by painting techniques. By contrast, advanced reel-to-reel film processing technologies yield very sophisticated film compositions, patterns and surface finishes not achievable by paint-based techniques. The team of Integument Technologies, Inc., the Johns Hopkins Center for Multifunctional Applique, and OptiMetrics, Inc has been working on the development of peel and stick fluoropolymer-based appliqués using advanced film technology. Our assumption is that appliqués may ultimately represent a paradigm shift in the way the military manages the interface between the equipment and its operating environment. Primarily, the focus of this Phase I project was to describe new developments related to an advanced surface barrier protection peel and stick appliqué system designed to include three dimensional camouflage patterns sublimed directly into the applique. The camouflage aspect of the applique is designed with a Phase II objective of achieving at least 20% signature reduction when incorporated onto large land vehicles and structures important to the U.S. Marine Corp. Further, this appliqué system will have both inherent and internally built-in capabilities for providing exceptional corrosion protection to all vehicles equipment and structures to which it will be applied.

The overall objective of the Phase I program was to demonstrate that dye sublimation technology combined with classic pigmentation can be used to create a 3D color palette within fluoropolymers that will enable a multi-color, patterned camouflage and corrosion control system suitable for use by the USMC. Highlights of the Phase I program include:

- **Successfully sublimed 4 absorption dyes within virgin ECTFE film in order to match Federal Color Code Mil Std. (1) Aircraft Black 37038, (2) Field Drab 33105, (3) Dark Green 34082, and (4) Sand 33303, demonstrating the potential of developing a broad color palette.**
- **Successfully sublimed dyes into films previously pigmented with “advanced effect” reflecting pigments. Pigments demonstrated the ability to brighten films and absorption dyes selectively darken them.**
- **Pigmented both glossy and low-gloss (matte finish) films, demonstrating the ability to meet wide ranging specular gloss requirements.**

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14. ABSTRACT The use of painted camouflage patterns on military hardware is a time-tested, cost-effective and practical countermeasure against human vision and aided vision target acquisition systems in many combat scenarios. However, the ability to integrate advanced patterns and optical effects is inherently limited by painting techniques. By contrast, advanced reel-to-reel film processing technologies yield very sophisticated film compositions, patterns and surface finishes not achievable by paint-based techniques. Currently the team of Integument Technologies, Inc., the Johns Hopkins Center for Multifunctional Applique, and OptiMetrics, Inc has been working on the development of peel and stick fluoropolymer-based appliques. Our assumption is that appliques may ultimately represent a paradigm shift in the way the military manages the interface between the equipment and its operating environment. Primarily, the focus of this project has been on new developments related to an advanced surface barrier protection peel and stick applique system designed to include three dimensional camouflage patterns infused directly into the applique.						
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- Successfully coated films with visibly-transparent coatings that reflect in the 2-20 micron range.
- Collected transmission and reflectance data on all films from 0.2 – 20 microns.
- Developed surface texturing model that confirmed the potential benefits of surface imprinting on film gloss at low grazing angles.

Outline of Final Report

1. 1st and 2nd generation optimization of sublimation of Mil STD Colors
2. Optical characterization of films
 - Specular gloss
 - Color determination
 - Transmission and Reflectance
3. Surface Texturing

1. Sublimation of Mil STD Colors

Military specification for coatings (MIL-C—46168D(ME)) along with FED-STD-595B color code were obtained for (1) matching MIL-STD colors to be incorporated into fluoropolymer films via Integument's sublimation processing, and (2) for determining appropriate testing (ASTM) protocols for measuring chromatic characteristics for sublimed films.

To date, a color palette of sublimed fluoropolymer films containing the following selected list (or sub-list) of colors as specified in Federal Color code 595B for camouflage has been performed.

- Aircraft Black 37038
- Field Drab 33105
- Dark Green 34082
- Sand 33303

These colors were 3-dimensionally sublimed into a 5 mil thick ECTFE fluoropolymer of three variations. The ECTFE films were varied as:

1. Film with matte finish
2. Film containing Afflairs 120 pigment : ~10% by volume (5-25 um mica, coated with TiO₂ (anatase) luster finish
3. Film containing Minatec 31 pigment: ~10% by volume (<15um mica, coated with SBSNO_x; non-luster finish

Using the results reported in the 2nd quarterly report we redefined our films to include pigmentation and matte finish surfaces. Based on last quarter's results we needed to

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demonstrate better specular gloss and color matching via the use of surface finishing and the inclusion of pigments into the ECTFE applique films.

In general, Integument's proprietary fluoropolymer sublimation process utilized anthraquinone based dyes that are sublimed directly into the fluoropolymer films using controlled temperature and pressure.

2. Optical characterization

The second generation sublimed films were optically characterized for specular gloss, and color matching as per ASTM testing specifications. All results for the gloss and color comparison to federal standard 595B are listed below. Included below are Tables that compare the Federal color code chip with the first generation and second generation of 3-dimensionally sublimed colored films.

SPECULAR GLOSS:

Method: ASTM D 523-89(99)

Conditions: Angle: 60°; samples backed with a black felt during testing

Note: All samples were referenced to Federal color code chips

TABLE 1
Specular Gloss (%) for 1st generation films
(Values are provided as the mean measured from 3 different samples)

Sample	Specular Gloss %
34082 (Fed. Color chip)	0.7
34082 clear film	71
34082 white pigmented film	64
37038 (Fed. Color chip)	0.8
37038 clear film	70
37038 white pigmented film	70
33303 (Fed. Color chip)	1.4
33303 clear film	69
33303white pigmented film	64
33105 (Fed. Color chip)	1.0
33105 clear film	64
33105white pigmented film	62

TABLE 2
Specular Gloss (%) for 2nd generation films
(Values are provided as the mean measured from 3 different samples)

Sample	Specular Gloss %
34082 (Fed. Color chip)	0.7
34082 unpigmented matte finish film	5.9
34082 pigmented Minatec 31	11
34082 pigmented Afflairs 120	14
37038 (Fed. Color chip)	0.8

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37038 unpigmented matte finish film	7.1
37038 pigmented Minatec 31	4.6
37038 pigmented Afflairs 120	7.3
33105 (Fed. Color chip)	0.9
33105 unpigmented matte finish film	9.7
33105 pigmented Minatec 31	8.5
33105 pigmented Afflairs 120	8.7

Results:

Table 1:

All of the first generation film samples (Table 1) had a high gloss reading typical of a gloss finish rather than the lusterless finish specified. The luster finish is strictly due to the ECTFE films which were not fabricated with matte (or designed surface texturing) or pigmentation. Optical values were obtained on both glossy transparent film and glossy white films. The white films were ECTFE pigmented (ca. 0.5% by weight) with TiO₂ pigments protected with organic inhibitors. The inhibitors were used in order to provide both UV absorbance as well as to eliminate any catalytic effect that the TiO₂ particles would have on both ECTFE film and dyes.

As predicted the sublimation of the dyes into glossy films had little effect on reducing the gloss or glare of the film coating system. The results listed in Table 1 were obtained in order to provide a baseline for subsequent studies that will investigate the effect on minimizing or eliminating gloss via the use of surface textured and pigmented films.

Table 2:

Based on the earlier results described above, three 2nd generation films were fabricated with the goal of reducing specular gloss. Three different film samples were fabricated, specifically:

1. The same ECTFE film as listed in Table 1 except that the new film was fabricated with a micro roughened surface to provide a matte finish
2. ECTFE containing Afflairs 120 pigment : ~10% by volume (5-25 um mica, coated with TiO₂ (anatase) luster finish without any surface texturing
3. ECTFE containing Minatec 31 pigment: ~10% by volume (<15um mica, coated with SBSNOx; non-luster finish without any surface texturing

The results listed in Table 2 show a substantially lower specular gloss for all of the 2nd generation films. Current specifications for military aircraft desire a specular gloss measure below 6.0 and this was achieved for Fed. Color Std. 34082 using the surface textured film and on Fed. Color Std. 37038 on the Minatec 31 pigmented ECTFE films. All of the other films show slightly higher specular gloss however it is obvious that various combinations of surface texturing and pigmentation can be coupled to provide

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any desired specular gloss requirement. This is a significant achievement which supports a successful Phase II program.

COLOR DETERMINATION

Method: ASTM D 2244-93(00) CIELAB Color Scale

Conditions: Illuminant C, 2° to the observer, Specular included BYK-Gardner TCS II, Model 8800 Instrument.

Note: Samples backed with white tile during testing

TABLE 3
1st generation films
Color Determination and Matching to Federal Color Code 595B

Sample	L*	a*	b*	ΔE^*_{ab}
34082 (Fed. Color chip)	36.85	-7.05	13.24	0
34082 clear film	33.06	5.22	1.42	17.45
34082 white pigmented film	40.10	-0.64	5.99	10.27
37038 (Fed. Color chip)	20.45	6.20	-7.36	0
37038 clear film	24.38	8.49	-3.38	6.06
37038 white pigmented film	27.31	1.96	0.23	11.07
33303 (Fed. Color chip)	61.63	-1.21	19.15	0
33303 clear film	54.68	2.18	14.25	9.15
33303 white pigmented film	68.77	0.07	6.17	14.86
33105 (Fed. Color chip)	41.14	1.57	20.16	0
33105 clear film	37.58	7.75	12.61	10.39
33105 white pigmented film	42.82	5.05	11.88	9.14

TABLE 4
2nd generation films
Color Determination and Matching to Federal Color Code 595B

Sample	L*	a*	b*	ΔE^*_{ab}
34082 (Fed. Color chip)	38.09	-6.70	12.98	0
34082 unpigmented matte finish film	39.19	-4.81	9.35	1.10
34082 pigmented Minatec 31	43.32	-2.61	2.17	5.23
34082 pigmented Afflairs 120	55.30	-1.84	-1.89	17.21
37038 (Fed. Color chip)	24.60	-4.24	4.06	0
37038 unpigmented matte finish film	26.74	0.20	6.47	2.14
37038 pigmented Minatec 31	32.76	1.57	-5.10	8.16
37038 pigmented Afflairs 120	57.32	0.20	-6.01	32.72
33105 (Fed. Color chip)	41.65	3.05	17.71	0
33105 unpigmented matte finish film	45.10	4.27	9.47	3.45
33105 pigmented Minatec 31	42.77	2.90	2.55	1.12
33105 pigmented Afflairs 120	69.61	1.61	2.92	27.96

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Results:

Description of values:

L^* - Measures colors as a function of light to dark wrt color, 0 = Dark; 100 = Light. Note: this does not refer to opacity, just the darkness of the color as specified in the Fed.

Color code MIL-STD.

a^* - Measures color as a function of Green to Red where Green is negative and Red is positive. 0 = 50/50 ratio of Green and Red.

b^* - Measures color as a function of Blue to Yellow where Blue is negative and Yellow is positive. 0 = 50/50 ratio of Blue and Yellow.

ΔE^*_{ab} - Equals the total deviation from the standard, i.e., the Fed color chip = 0 (no color deviation). Note that in the automotive industry a value greater than 3 is unacceptable and that values greater than 10 are discernible by the human eye.

Based on the results in Table 3 (1st generation of films) we find that the sublimed colors differ by 10 ΔE^*_{ab} units on average. These values are calculated from the differences measured for a^* and b^* from the standard federal color code chip. It is important to note that the color deviation can be drastically changed by using different backgrounds. This is due to the fact that the sublimed ECTFE films were not opaque and thus the measurements were greatly affected depending on the color of the backing material used during the measurement. In the cases listed in Table 3 and 4 all of the samples were backed with a white tile.

The 1st generation film results listed in Table 3 begged the question as to whether or not the deviations in Fed Color Stds. were problematic. Based on the results obtained on the 2nd generation films listed in Table 4 the answer at this point is no. For each Fed Color Std. we were able to demonstrate values less than 3 (on at least 1 type of fabricated film) which are acceptable results. In addition, it is clear that via the added surface texturing and/or incorporation of pigments we can adjust the 3-dimensional color characteristics in order to meet the required standards.

The goal of this section of the Phase I project was to demonstrate the effective ability to sublime a color into the ECTFE applique films and then to measure the basic color characteristics in order to begin developing a color palette of applique films to be used for fabricating various camouflage designs. Based on both Specular gloss and Color Determination tests, we have demonstrated the capability to provide a color palette consisting of matched Federal Color Stds.

In order to achieve our desired results we need to begin modifying our processing in order to optimize well defined colors (and methods for reproducing them) sublimed into ECTFE based applique films that have various additives (e.g., pigments) and surface textures that can influence surface and bulk transmission and reflection properties. At this

initial stage in the game we now know that introduction of pigments and surface texturing for achieving novel optical properties (e.g., BRDF) will also influence the color of the sublimed dyes and can be used effectively to provide desired colors and subsequent patterns that effectively minimize detection. This is important information since these results demonstrate the ability to successfully develop a unique applique system that will be defined in the Phase II proposal. Additives and surface texturing will ultimately provide the optical reflectance characteristics that will determine the efficacy of these applique systems for providing required high performance camouflage properties.

Transmission and Reflectance Results

Besides the direct optical comparison to values associated with conventional camouflage paints other more fundamental optical physics related to transmission and reflectance in various regions of the electromagnetic spectrum were obtained. This was necessary in order to demonstrate a difference between conventional paint camouflage technology and Integument's 3-dimensional camouflage applique technology. Specifically, the data below will provide the effects of adding dyes, pigments, and surface top coatings to the applique films. The importance in incorporating and testing a camouflage design is obviously underscored however it is imperative that the applique sublimation palette be characterized as to why it might out perform or provide better potential than paint.

More precisely, consider illustrations in Figure 1.

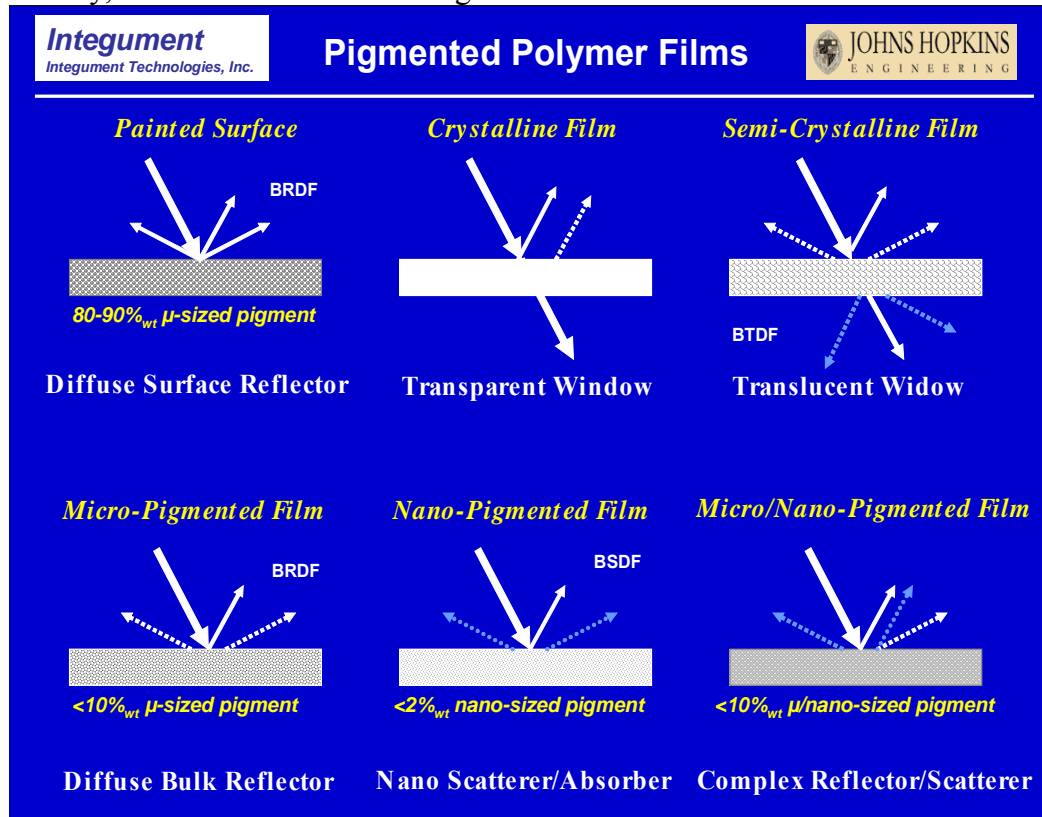


FIGURE 1

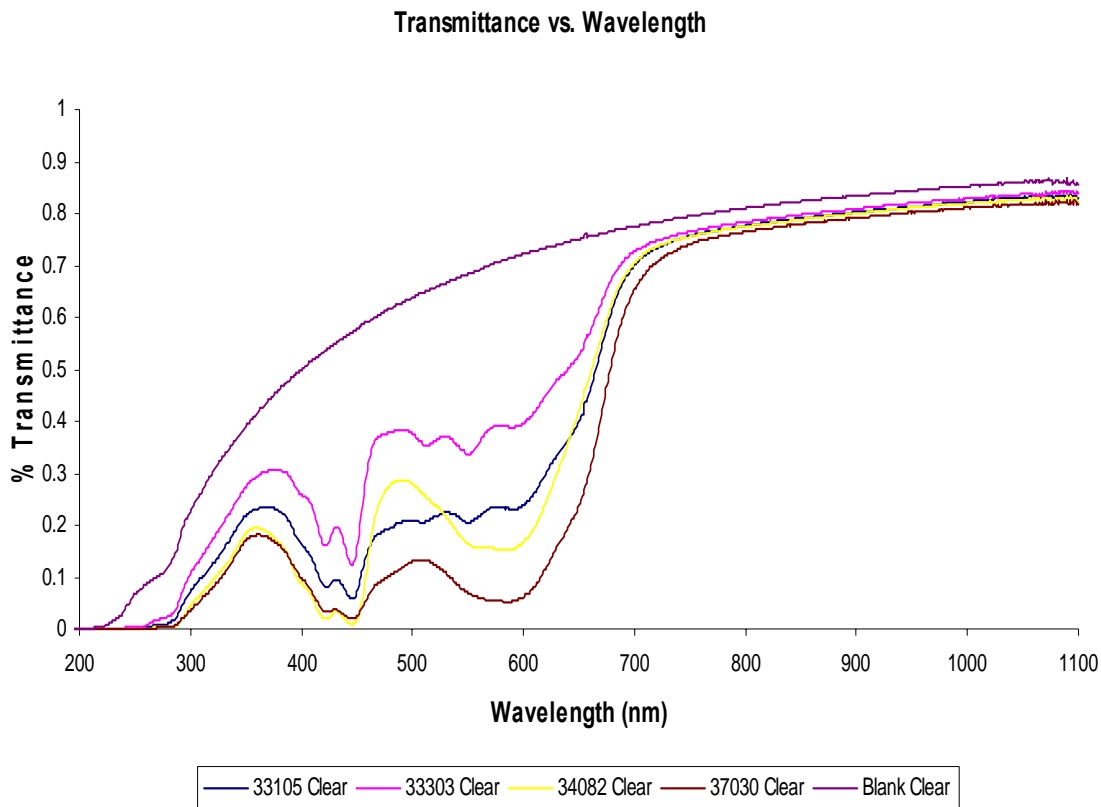
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Figure 1 illustrates six different kinds of materials and their associated reflectance/scattering characteristics. Assuming a smooth surface, a traditional painted surface will be comprised of roughly 80-90% solids that have geometries in the range of microns. This acts as a diffuse surface reflector such that one observes reflectance at almost any angle of observation, in particular at low angles based on a Bidirectional Reflectance Distribution Function (BRDF). Even in cases that the pigment is a good absorber of incoming radiation such that specular reflectivity is attenuated, the BRDF characteristics of this system results in high reflectivity at low angles of observation.

Alternatively, a purely crystalline polymer or (or plastic film) has significantly different properties such that radiation scattered from the surface and bulk is significantly attenuated and most is transmitted creating a transparent window. Thus, from an initial starting point one can see the major difference between a painted surface and a polymeric material with respect to attenuating reflectance of light. Specifically, a polymeric starting material potentially provides a coating system (e.g. applique) that has reflective characteristics that fall outside the Bidirectional Reflectance Distribution Function (BRDF) associated with pigmented paint coating systems.

Transmission and Reflectance Results

Figure 2



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Figure 2 shows the absorbance spectrum of clear ECTFE 5 mil films sublimed with the four chosen MIL STD colors as listed previously in this report. The major observation is that the 3 dimensionally sublimed dyes only absorb in the visible 0.3um to 0.7um region thus influencing only the visible portion of the light spectrum.

Figure 3

Clear Total Reflectance

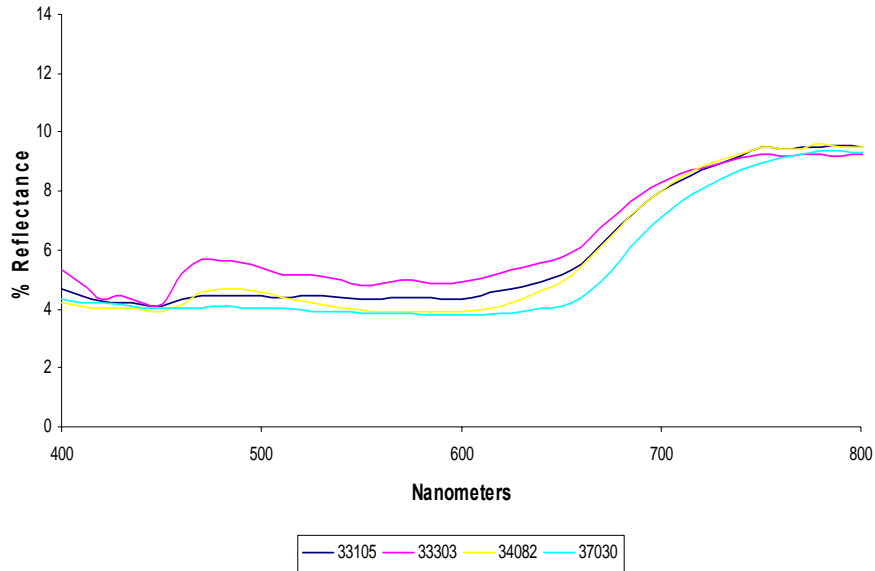
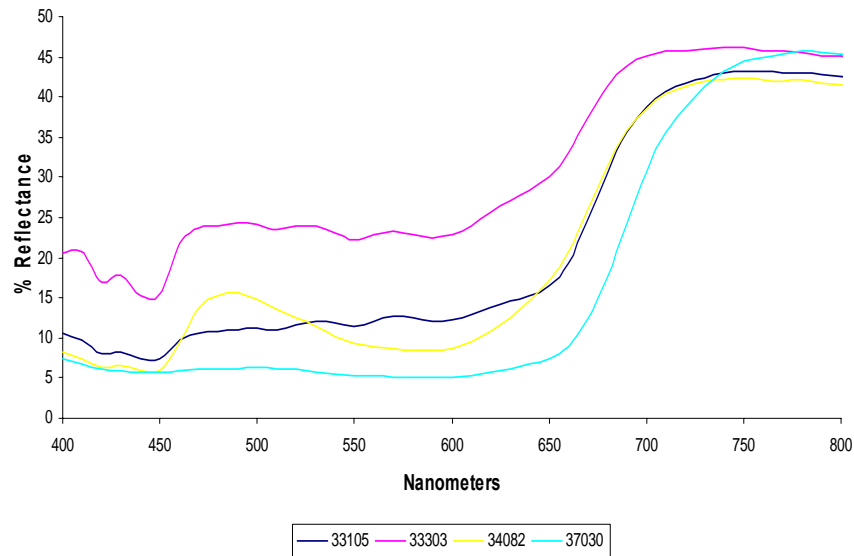


Figure 3 illustrates the reflectance characteristics of the same films listed in Figure 2 (i.e., clear sublimed ECTFE). The reflectance data shows very little reflectance (2% -10%) indicating that most of the light in the visible region of the spectrum is either absorbed or passed through the films.

Figure 4

White Total Reflectance



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Alternatively, Figure 4 illustrates the effect of adding a TiO_2 pigment into the clear film. The added pigment acts to increase the film reflectance slightly in the 0.4 μm to 0.6 μm region (i.e., it tends to brighten the sublimed dyes) with a drastic increased reflectance above 0.7 μm .

This is better illustrated in Figure 5 where we plot the Total Reflectance (0.4 μm to 2.5 μm) of a dye (Fed. Color STD. 33303 Sand) sublimed clear ECTFE versus a dye sublimed pigmented ECTFE film. The results demonstrate that sublimed dyes have very little effect on reflectivity and only add visible color to the applique films. The addition of a pigment however, provides absorption/reflectance properties from 0.4 μm to 2.3 μm region of the light spectrum. Thus, dyes and pigments can be used together to achieve various optical properties of applique films.

For example Figure 6 illustrates the various capabilities that can be incorporated into applique materials for achieving advanced effects coupled with the patterning capability of Integument's Dye processing .

Figure 5

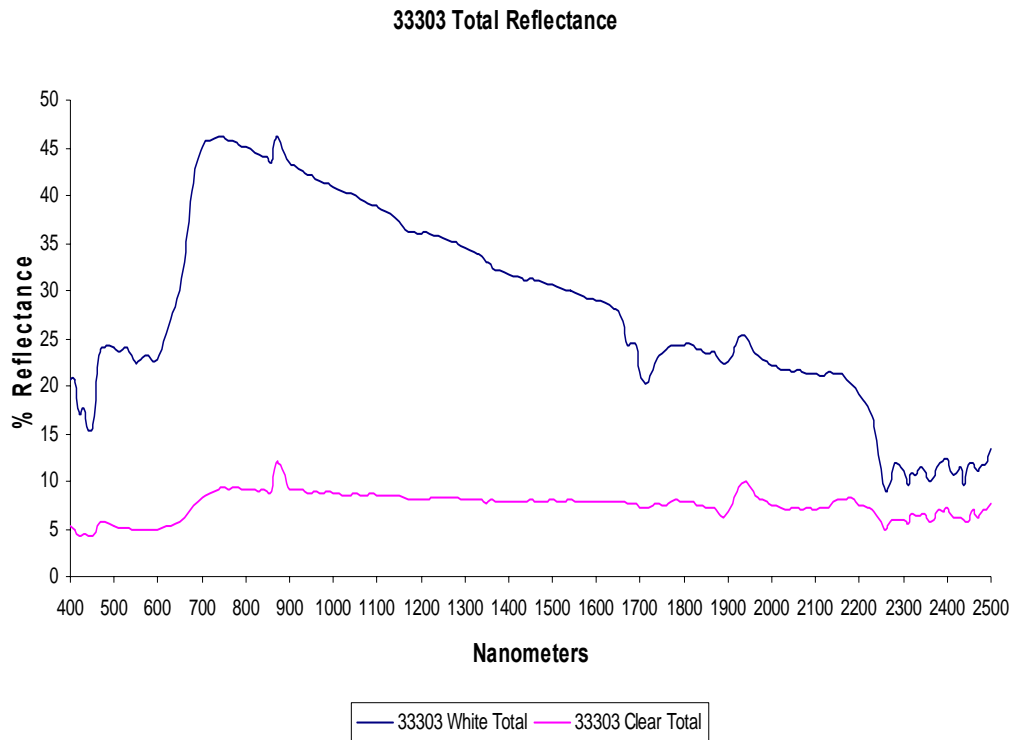
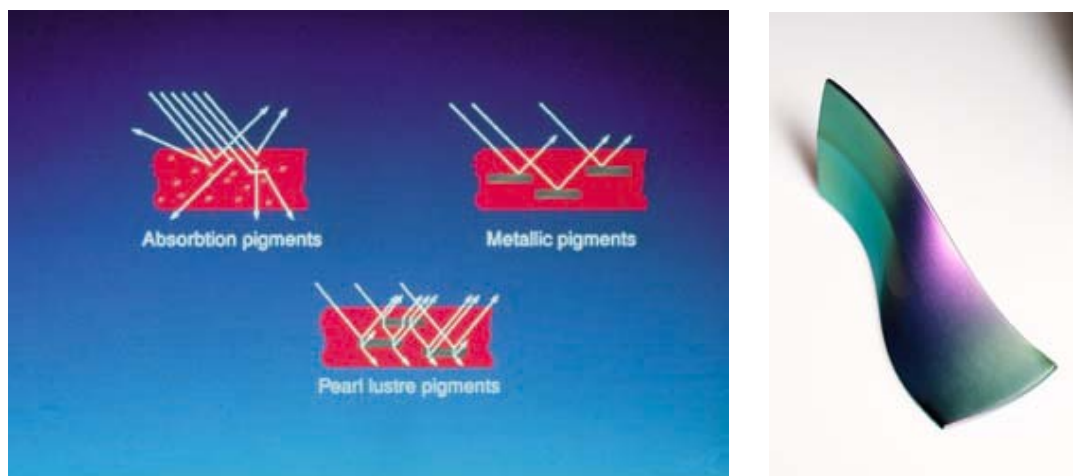


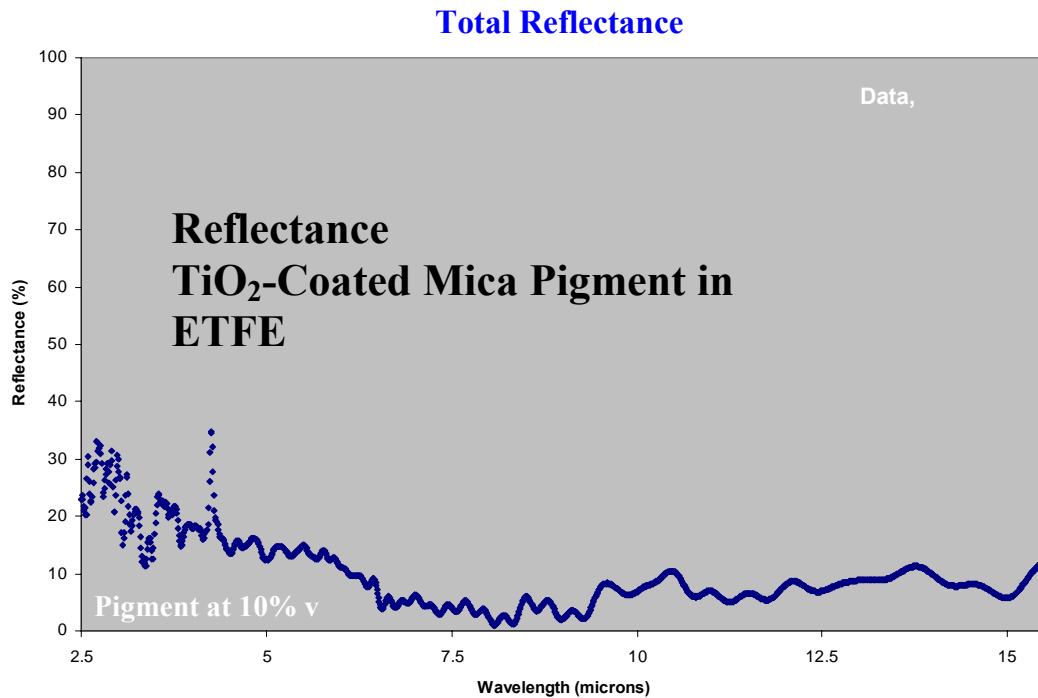
Figure 6



Sophisticated flake pigments (mica, tabular alumina, bismuth oxychloride, etc.) are now available to introduce or accent functionality into plastics at levels not previously achievable.

In addition to investigating the Transmission and Reflectance characteristics in the 0.4 μ m to 2.5 μ m region of the light spectrum we also measured the reflectance properties of the TiO₂ pigmented films in the 2.5 μ m to 15 μ m region. Figure 7 shows that neither dye nor pigments have any effect and the applique materials have virtually zero reflection characteristics in light spectrum regions over 2.5 μ m.

Figure 7



In order to affect regions of the light spectrum that lie above 2.5um one would have to use a top coating on the applique. For example Figure 8 shows that a of top coating of Indium Tin Oxide (ITO) on the polymer Polyester terephthalate (PET) has little affect on surface reflectivity in the 0.4um thru 2.5um region of the light spectrum. However, investigation of the ITO affect on regions above 2.5um show significant enhancement as illustrated in Figure 9. During the last two weeks of this Phase I project we have successfully demonstrated the ability to coat our ECTFE applique film with vapor sputtered ITO. The surface resistance of these films were measured to be ~ 100 ohms per square however, we did not have the time to optically characterize these materials. These films are available upon request.

Figure 8

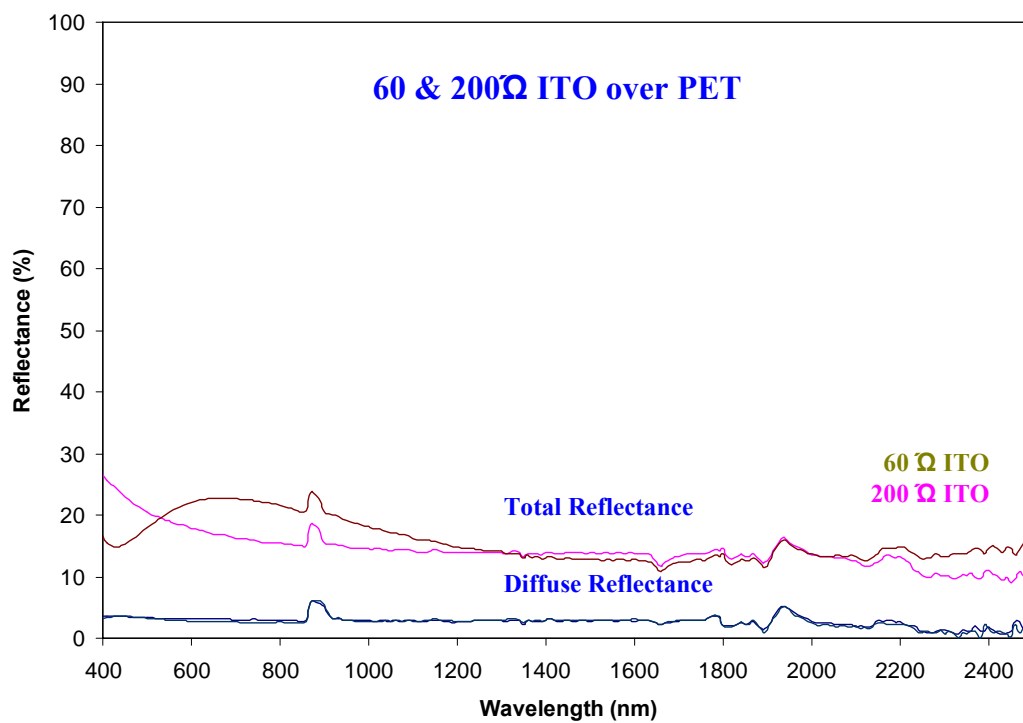
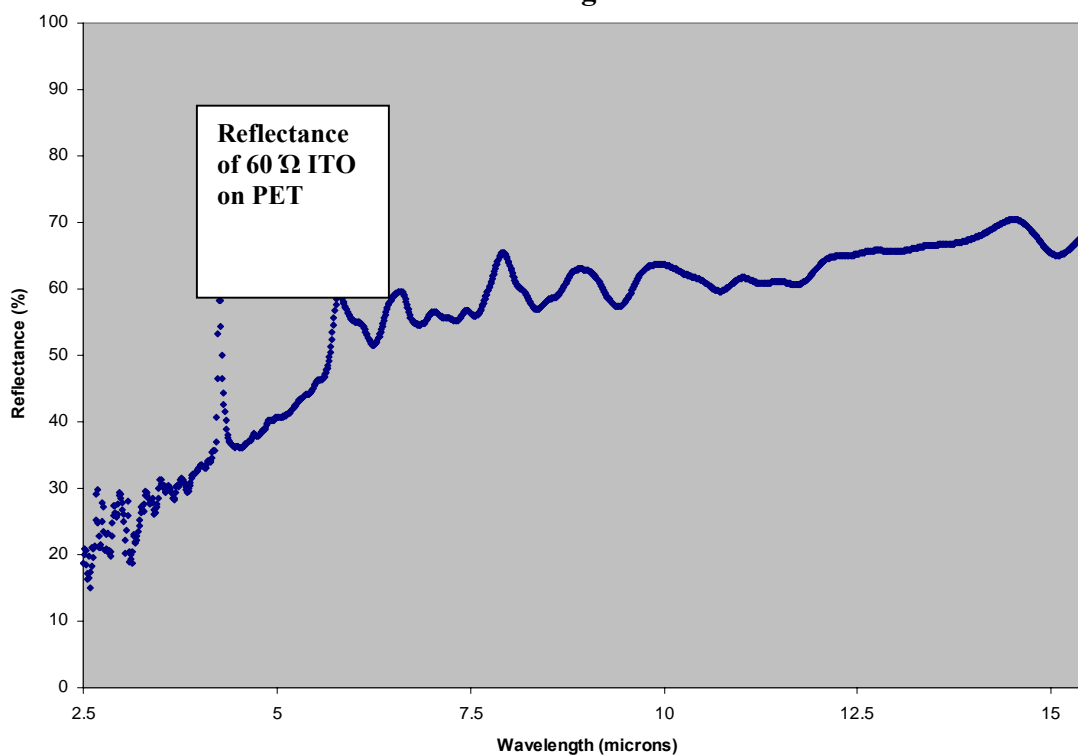
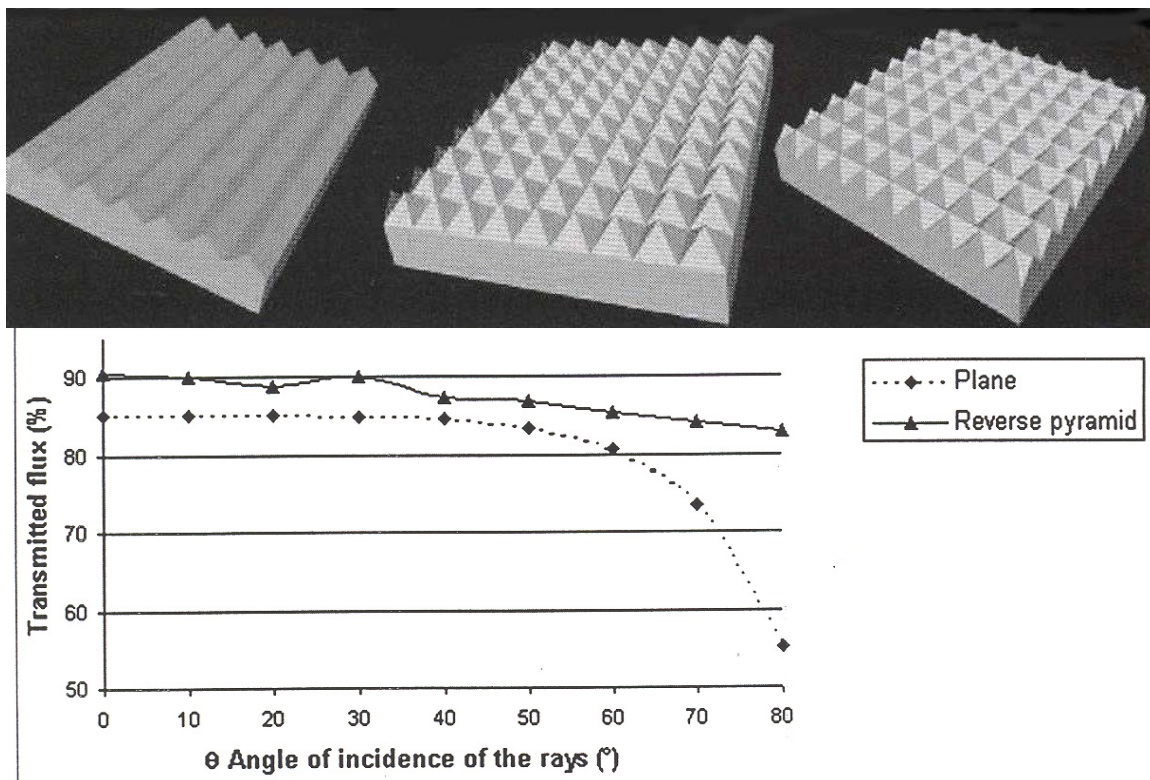


Figure 9



3. Surface Texturing: Finally, the Phase I program included preliminary modeling and design of potential surface geometries that can be utilized to control glint, glare and other optical characteristics of the applique films. The team has discussed various surface features modeled by OptiMetrics, Inc. that will have the optimal effect with regards to providing the low angle spectral reflectance that may demonstrate the advantage applique film has over conventional paint camouflage designs. For example, Figure 10 illustrates the comparison of a flat surface versus a textured surface geometry as a function of transmitted visible light versus the angle of incidence. Clearly the reverse pyramid textured surface provides less reflection at higher angles of incidence thus demonstrating the control of surface optics using specific surface textures and geometries geometries.

FIGURE 10



In addition, Figure 11 shows the average surface reflectance of a film having a saw-toothed pattern as a function of surface rounding or aspect ratio.

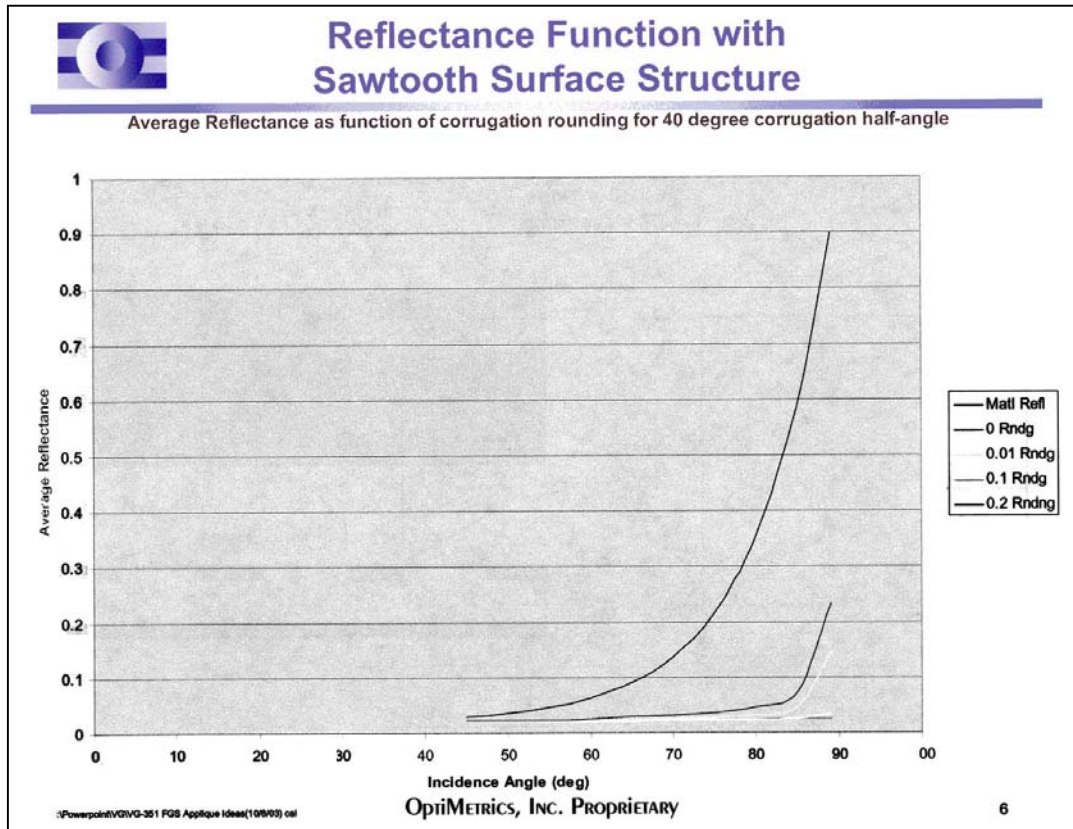
In general, our model indicates that for a fluoropolymer based material the aspect ratio (i.e., height to width) is important along with the half angle of the saw-tooth which needs to be 38.5° in order to achieve total internal reflection. In addition, the dimensionality of the geometry is unimportant i.e., the shape can be triangular or pyramidal. The geometry height is not important as long as the aspect ratio is maintained. Finally, as shown in

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Figure 11 the degree of surface rounding of the top of the triangle or pyramid is critical and thus, our tooling will need to be developed for this capability.

Note that the top line in Figure 11 represents a smooth surface having ca. 90% reflectance whereas the bottom line with 0.0 rounding has < 0.1% reflectance. The other curves represent the fall off of internal reflectance as a function of rounding of the saw-tooth features.

Figure 11



Conclusions:

The Results of the Phase I program have successfully:

1. Demonstrated the ability to sublime 3-D colors into a fluoropolymer based applique film.
2. Demonstrated the ability to add pigments in conjunction with the dyeing process

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- 3. Demonstrated the ability to apply a conductive transparent ITO topcoat to the applique films**
- 4. Demonstrated the ability to control both Specular Gloss and Match Federal STD. MIL colors**
- 5. Demonstrated Absorbance and Reflectivity characteristics of:**
 - a. Sublimed Dyes**
 - b. Pigments**
 - c. Top Coatings**
- 6. Developed a preliminary surface geometry model for controlling applique optical characteristics.**

Based on the results listed above the team is in excellent position to continue the development and final fabrication of a unique innovative camouflage applique during a Phase II program. Within a Phase II program the team will continue:

- 1. The development and optimization of a color palette via dye sublimation**
- 2. The development of appliqués having advanced pigmentation**
- 3. The development of surface texturing using surface geometry models**
- 4. Top Coating optimization**
- 5. Signature reduction testing**
 - a. Laboratory**
 - b. Field trials**